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Synthesis, Spectroscopic Characterization, and in vitro Biological Activity of Organotin(IV) Complexes of (*E*)-3-(4-Methoxyphenyl)-2-phenyl-2-propenoic Acid

Sadiq-ur-Rehman,[1] Khadija Shahid,[1] Saqib Ali,[1] Mohammad Mazhar,[1] Amin Badshah,[1] George Eng,[2] Xueqing Song,[2] and Janusz Ryczkowski[3]

[1]*Department of Chemistry, Quaid-i-Azam University, Islamabad-45320, Pakistan*

[2]*Department of Chemistry & Physics, University of the District of Columbia, Washington DC, 20008, USA*

[3]*Department of Chemical Technology, Faculty of Chemistry, University of Maria Curie-sklodowska, P1. M. Curie-sklodowskiej 3, 20-031 Lublin, Poland*

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ABSTRACT: *Organotin(IV) complexes with general formula $R_{4-n}SnL_n$ have been synthesized by the reaction of tri- and diorganotin chlorides with sodium salt of (E)-3-(4-methoxyphenyl)-2-phenyl-2-propenoic acid in dry toluene, where R=CH_3, C_2H_5, n-C_4H_9, C_6H_5, n = 1 or 2, and L = [$\bar{O}OCC(C_6H_5)$ $CH(C_6H_4OCH_3)$]. Dimeric distannoxanes, [$(R_2SnOOCC(C_6H_5)CH(C_6H_4OCH_3)_2O]_2$, were synthesized by the reaction of acid with diorganotin oxides in 1:1 molar ratio. Multinuclear NMR (^1H, ^{13}C, ^{119}Sn), infrared, mass, and Mössbauer spectral techniques have been used to ascertain their structures in solution and as solid. The spectroscopic results showed that triorganotin(IV) derivatives are 4-coordinated monomers in noncoordinating solvents and 5-coordinated polymers with bridging carboxylate groups in the solid state. However, diorganotin and dimeric compounds are 5-coordinated in noncoordinating solvents while in the solid phase may have higher coordination. Biological screening tests showed that most of the compounds have significant activity against different bacteria and fungi.* © 2005 Wiley Periodicals, Inc. Heteroatom Chem 16:175–183, 2005; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20089

Correspondence to: Saqib Ali; e-mail: drsa54@yahoo.com.
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INTRODUCTION

Organotin compounds have important applications in several areas and hence are made industrially on a large scale. By far, the major use is in the stabilization of PVC to prevent thermal degradation during processing and long-term photo degradation [1]. Dialkyltin derivatives are the additives of choice for this purpose, e.g. dioctyltin compounds are used in PVC for food packing, drink bottles, and potable water piping [2]. Dibutyltin dilaurate is widely employed as a catalyst for the production of polyurethane foams or for the room temperature curing of silicone rubber [3]. Diorganotin carboxylates are also reported to have antitumor activity [4,5]. However, triorganotins have biocidal properties [6] and are used as pesticides or insecticides in agriculture [7,8].

Because of economic context and our continuous interest in the synthesis, structural, spectroscopic and biocidal studies of organotin carboxylates [9–11], we have synthesized a series of new organotin(IV) carboxylates. The structures of these compounds have been investigated by infrared, multinuclear NMR (^{1}H, ^{13}C, ^{119}Sn), Mössbauer spectroscopies, and mass spectrometry. Biological testing has been performed against various bacteria and fungi. Most of the compounds were found to have significant biological activity.

RESULTS AND DISCUSSION

All the tri- and diorganotin complexes of (*E*)-3-(4-methoxyphenyl)-2-phenyl-2-propenoic acid (see Fig. 1) were synthesized in dry toluene by refluxing the mixture of sodium salt of acid and corresponding organotin chloride in 1:1 and 2:1 molar ratio, respectively (Eqs. (1) and (2)).

$$R_3SnCl + NaL \longrightarrow R_3SnL + NaCl \quad (1)$$

R = Me(**II**), *n*-Bu(**IV**), Ph(**VI**)

$$R_2SnCl_2 + 2NaL \longrightarrow R_2SnL_2 + 2NaCl \quad (2)$$

R = Me(**Ia**), *n*-Bu(**IIIa**), Et(**Va**)

Whereas dimeric distannoxanes were prepared on treating (*E*)-3-(4-methoxyphenyl)-2-phenyl-2-propenoic acid with diorganotin oxide in 1:1 molar ratio, using Dean and Stark apparatus.

$$4R_2SnO + 4LOH \longrightarrow [(R_2SnL)_2O]_2 + 2H_2O \quad (3)$$

R = Me(**Ib**), *n*-Bu(**IIIb**), Et(**Vb**)

The complexes are quite stable and soluble in common organic solvents. The physical data are reported in Table 1.

Infrared Spectra

The infrared spectra of the sodium salt, ligand, and organotin complexes have been recorded as KBr disks in the range 4000–400 cm^{-1}. The important bands that indicate formation of complexes are ν(COO), ν(Sn—C), ν(Sn—O), and ν(Sn—O—Sn) which are given in Table 2.

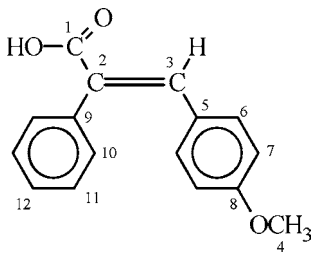


FIGURE 1 Numbering scheme and structure of (*E*)-3-(4-methxoyphenyl)-2-phenyl-2-propenoic acid.

Attempts have been made to correlate the values of characteristic vibrations with their precursors and literature reports to predict structures of the synthesized compounds [12]. The absence of the broad band for ν(O—H) in the range 2400–3400 cm^{-1} in the spectra of the sodium salt and organotin(IV) derivatives shows the deprotonation of the ligand acid. Moreover, absorption bands in the range 515–559 cm^{-1} and 427–479 cm^{-1} due to Sn—C and Sn—O bonds, respectively, confirm further the formation of products. The ν(COO) stretching vibrations are related with bonding mode of the ligand [13]. The $\Delta\nu$ values [$\Delta\nu = \nu_{asym}(COO) - \nu_{sym}(COO)$] are comparable to the sodium salt that indicate the bidentate nature of the ligand [14,15].

The IR spectra of dimeric tetraorganodicarboxylato stannoxanes are almost similar to those observed for diorganotin dicarboxylates with exception of absorption bands found in the range 697–706 cm^{-1}, characteristic for Sn—O—Sn—O ring in these compounds [16,17].

Multinuclear NMR

The ^{1}H and ^{13}C NMR data are shown in Tables 3–6. The important resonances are assigned by their multiplicity and intensity pattern, integration, and coupling constants.

The aromatic carbon resonances (both in the ligand and phenyltin, **VI**) were assigned by comparison of the chemical shifts with those calculated from incremental method [18], while aliphatic proton and carbon resonances were assigned on comparison with the literature values [19]. In triphenyltin(IV) derivative (compound **VI**), a complex pattern is observed in the range of 6.72–7.44 ppm due to the aromatic protons of the ligand and phenyl groups. In triorganotins, the coupling constant is observed only for trimethyltin derivative, $^2J[^{117/119}Sn-^1H]$ = 56, 58 Hz, that suggests a tetrahedral geometry around the tin atom and is further supported by the C—Sn—C bond angle 111.0°, calculated by Lockhart's equation [20]. The values of $^1J[^{117/119}Sn-^{13}C]$ (Table 5) and bond angles (C—Sn—C) 111.7° and 110.9°, enumerated from ^1J values [20,21] for trimethyltin and tri-*n*-butyltin derivatives, respectively, also confirm the proposed tetrahedral structure in noncoordinating solvent. Similarly, the values of δ ^{119}Sn (Table 7) are also consistent with earlier reports, describing tetrahedral geometry [22,23].

TABLE 1 Physical Data of the Synthesized Organotin(IV) Derivatives of (*E*)-3-(4-Methoxyphenyl)-2-phenyl-2-propenoic Acid[a]

Comp. No.	*Compound (Formula Weight)*	*MP (°C)*	*Yield (%)*	*%C Calc. (Found)*	*%H Calc. (Found)*
Ia	$Me_2SnL_2C_{34}H_{32}O_6Sn$ (655)	168–170	80	62.29 (62.30)	4.89 (4.86)
Ib	$[(Me_2SnL)_2O]_2C_{72}H_{76}O_{14}Sn_4$ (1640)	151–153	70	52.68 (52.74)	4.63 (4.85)
II	$Me_3SnLC_{19}H_{22}O_3Sn$ (417)	116–118	90	54.68 (54.38)	5.28 (5.23)
IIIa	n-$Bu_2SnL_2C_{40}H_{44}O_6Sn$ (739)	118–120	80	64.95 (64.53)	5.95 (6.05)
IIIb	$[(n\text{-}Bu_2SnL)_2O]_2C_{96}H_{124}O_{14}Sn_4$ (1976)	103–105	75	58.30 (58.37)	6.28 (6.30)
IV	n-$Bu_3SnLC_{28}H_{40}O_3Sn$ (543)	108–110	85	61.88 (61.53)	7.37 (7.25)
Va	$Et_2SnL_2C_{36}H_{36}O_6Sn$ (683)	140–143	82	63.25 (63.35)	5.27 (5.30)
Vb	$[(Et_2SnL)_2O]_2C_{80}H_{92}O_{14}Sn_4$ (1752)	130–132	82	54.79 (54.56)	5.25 (5.16)
VI	$Ph_3SnLC_{34}H_{28}O_3Sn$ (603)	328–330	70	67.66 (67.86)	4.64 (4.73)

[a]In all other tables the formulation and number of the compounds are the same as given in this table.

TABLE 2 Infrared Data (cm^{-1}) of Organotin(IV) Derivatives of (*E*)-3-(4-Methoxyphenyl)-2-phenyl-2-propenoic Acid

	$\nu(COO)$					
Comp. No.	*Asym.*	*Sym.*	$\Delta\nu$	$\nu(Sn\text{–}C)$	$\nu(Sn\text{–}O)$	$\nu(Sn\text{–}O\text{–}Sn)$
Ia	1601 s	1418 m	183	546 m	460 w	–
Ib	1604 s	1414 m	190	559 m	479 w	706 s
II	1601 s	1420 s	181	556 m	427 w	–
IIIa	1601 s	1418 m	183	520 w	429 w	–
IIIb	1605 s	1416 s	189	559 m	474 m	702 s
IV	1603 s	1418 m	185	550 w	464 w	–
Va	1604 s	1420 m	184	515 w	453 m	–
Vb	1608 s	1415 m	193	558 s	478 w	697 s
VI	1570 s	1403 s	167	–	496	–
HL(acid)	1671 s	1420 s	251	–	–	–
NaL	1605 s	1389 s	216	–	–	–

s = Strong; m = medium; w = weak.

TABLE 3 ^{1}H NMR Data[a,b,c] of Triorganotin(IV) Derivatives of (*E*)-3-(4-Methoxyphenyl)-2-phenyl-2-propenoic Acid

^1H No.	(**HL**) *Acid*	(**II**) *Me_3SnL*	(**IV**) *n-Bu_3SnL*	(**VI**) *Ph_3SnL*
3	7.64 (s)	7.84 (s)	7.89 (s)	7.62 (s)
4	3.49 (s)	3.77 (s)	3.69 (s)	3.72 (s)
6	6.76 (dd, 8.8, 2.5)	7.01(dd, 9.1, 2.7)	7.05 (d, 8.9)	6.89 (d, 8.5)
7	6.43 (dd, 8.8, 2.5)	6.69 (dd, 8.8, 3.1)	6.67 (d, 8.9)	6.61 (d, 8.5)
10	7.12 (m)	7.31–7.43 (m)	7.35–7.41 (m)	7.36–7.44 (m)
11	6.98–6.99 (m)	7.25–7.28 (m)	7.29–7.32 (m)	7.19–7.25 (m)
12	6.98–6.99 (m)	7.25–7.28 (m)	7.29–7.32 (m)	7.08–7.11 (m)
α	–	0.62 (s); ^2J[56,58]	1.66–1.75 (m)	–
β	–	–	1.43–1.49 (m)	6.72–6.78 (m)
γ	–	–	1.33–1.39 (m)	6.72–6.78 (m)
δ	–	–	0.99 (t, 7.3)	–

[a]Chemical shift (δ) in ppm. ^2J[^{119}Sn$-^1$H] and ^3J(^{1}H$-^1$H) in Hz are listed in square brackets and parenthesis, respectively. Multiplicity is given as s = singlet, d = doublet, dd = doublet of doublet, m = multiplet.
[b]Numbering is according to Fig. 1.
[c]

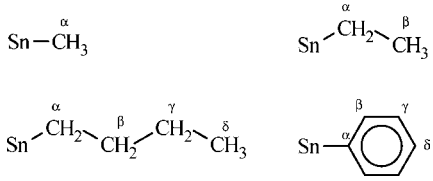


TABLE 4 ^{1}H NMR Data[a,b,c] of Diorganotin(IV) of (*E*)-3-(4-Methoxyphenyl)-2-phenyl-2-propenoic Acid "a" and "b"

^{1}H No.	(**Ia**) *Me_2SnL_2*	(**Ib**) *$[(Me_2SnL)_2O]_2$*	(**IIIa**) *$n\text{-}Bu_2SnL_2$*	(**IIIb**) *$[(n\text{-}Bu_2SnL)_2O]_2$*	(**Va**) *Et_2SnL_2*	(**Vb**) *$[(Et_2SnL)_2O]_2$*
3	8.02 (s)	7.73 (s)	8.01 (s)	7.68 (s)	8.03 (s)	7.72 (s)
4	3.79 (s)	3.78 (s)	3.79 (s)	3.75 (s)	3.79 (s)	3.75 (s)
6	7.07 (d, 8.6)	7.07 (d, 8.6)	7.07 (d, 8.5)	7.0 (d, 8.4)	7.08 (d, 8.7)	7.01 (d, 8.5)
7	6.73 (d, 8.6)	6.73 (d, 8.6)	6.72 (d, 9.0)	6.69 (d, 9.8)	6.73 (d, 8.7)	6.68 (d, 8.5)
10	7.41–7.43 (m)	7.29–7.31 (m)	7.39–7.44 (m)	7.39–7.44 (m)	7.38–7.44 (m)	7.38–7.44 (m)
11	7.41–7.43 (m)	7.29–7.31 (m)	7.29–7.31 (m)	7.29–7.31 (m)	7.29–7.32 (m)	7.29–7.32 (m)
12	7.22 (m)	7.22 (m)	7.18–7.21 (m)	7.18–7.21 (m)	7.19–7.23 (m)	7.19–7.23 (m)
α	1.08 (s); ^{2}J[82.5]	0.773 (s), 0.617 (s)	1.74–1.76 (m)	1.58–1.61 (m)	1.69–1.78 (q, 7.6)	1.64 (b.s)
β	–	–	1.41–1.44 (m)	1.36–1.38 (m)	1.35–1.41 (t, 7.7)	1.23–1.29 (t, 6.98)
γ	–	–	1.29–1.33 (m)	1.27–1.30 (m)	–	–
δ	–	–	0.94 (t, 7.3)	0.88 (t, 7.2)	–	–

[a]Chemical shifts (δ) in ppm. $^{2}J[^{119}Sn-^{1}H]$; $^{3}J[^{1}H-^{1}H]$ in Hz are listed in parenthesis. Multiplicity is given as s = singlet, m = multiplet, and b.s = broad signal.
[b]Numbering is according to Fig. 1.
[c]See footnote "c" of Table 3 for α, β, γ, δ.

For diorganotin dicarboxylates as in compound **Ia**, the value of $^{2}J[^{119}Sn-^{1}H] = 82.5$ Hz indicates penta- or hexa-coordinated complex. The ^{119}Sn chemical shift values for diorganotin derivatives fall in the range from −90 to −190 ppm, showing pentacoordinated systems [24] as proposed in Fig. 2d. However, tetraorganodicarboxylato stannoxanes (**Ib**, **IIIb**, and **Vb**) may adopt a characteristic dimeric structural mode, Fig. 2f [23]. The two sets of signals in the ^{1}H and ^{13}C NMR spectra for R groups attached with the endo- and exo-cyclic tin atoms confirm this assessment. Furthermore, two signals observed in the ^{119}Sn NMR spectra also reflecting different environment around the tin atoms in the same molecule [12,23].

Mössbauer Spectra

To explore further the structure of these compounds and behavior of ligand toward tin atom, Mössbauer and ^{119}Sn NMR spectra were also recorded for some complexes. Mössbauer parameters along with δ ^{119}Sn values for the representative compounds are given in Table 7. The quadrupole splitting (Δ) 3.80 mms^{-1} and isomer shift 1.41 mms^{-1} for **II** show a polymeric trans-R_3SnO_2 arrangement with bridging

TABLE 5 ^{13}C NMR Data[a,b,c] of Triorganotin(IV) Derivatives of (*E*)-3-(4-Methoxyphenyl)-2-phenyl -2-propenoic Acid

^{13}C No.	(**HL**) *Acid*	(**II**) *Me_3SnL*	(**IV**) *$n\text{-}Bu_3SnL$*	(**VI**) *Ph_3SnL*
1	173.03	173.20	172.57	173.80
2	129.03	131.63	131.80	128.46
3	142.16	139.36	138.72	144.26
4	55.23	55.16	54.71	55.15
5	126.90	127.75	127.62	126.93
6	132.73	132.10	131.79	130.45
7	113.75	113.55	113.30	113.39
8	160.62	159.85	159.63	159.87
9	135.76	137.34	137.55	136.66
10	128.81	128.53	128.16	127.43
11	129.79	129.82	129.57	128.02
12	127.91	127.32	126.96	127.79
α	–	−2.16 (380.4, 398)	16.41 (348, 362)	140.68
β	–	–	27.69	135.89
γ	–	–	26.81 (62)	132.40
δ	–	–	13.49	132.69

[a]Chemical shifts (δ) in ppm. ^{n}J ($^{117/119}Sn-^{13}C$) and $^{n}J(^{119}Sn-^{13}C)$ in Hz are listed in parenthesis.
[b]Numbering is according to Fig. 1.
[c]See footnote "c" of Table 3 for α, β, γ, δ.

TABLE 6 ^{13}C NMR Data[a,b,c] of Diorganotin(IV) of (*E*)-3-(4-Methoxyphenyl)-2-phenyl-2-propenoic Acid "a" and "b"

^{13}C No.	(**Ia**) *Me_2SnL_2*	(**Ib**) *$[(Me_2SnL)_2O]_2$*	(**IIIa**) *$n\text{-}Bu_2SnL_2$*	(**IIIb**) *$[(n\text{-}Bu_2SnL)_2O]_2$*	(**Va**) *Et_2SnL_2*	(**Vb**) *$[(Et_2SnL)_2O]_2$*
1	177.65	174.02	177.47	174.28	177.66	174.43
2	132.19	129.42	136.60	133.70	132.08	129.61
3	141.77	136.33	141.32	136.33	141.44	136.55
4	55.20	55.20	55.20	55.20	55.19	55.20
5	127.19	127.19	127.33	127.17	127.27	127.13
6	132.51	132.51	132.46	132.01	132.47	132.47
7	113.70	113.70	113.67	113.67	113.68	113.61
8	160.39	159.84	160.29	159.76	160.33	159.82
9	138.36	137.54	138.21	137.89	138.41	137.84
10	128.78	128.78	128.77	128.57	128.78	128.78
11	129.74	129.74	129.72	129.61	129.69	129.69
12	127.77	127.77	127.91	127.70	128.54	127.71
α	4.87	8.64, 7.47	25.19	28.43, 27.31	17.72	20.90, 20.67
β	–	–	26.86	27.74, 26.92	9.03	10.05, 9.61
γ	–	–	26.32	26.73	–	–
δ	–	–	13.66	13.77	–	–

[a]Chemical shifts (δ) in ppm.
[b]Numbering is according to Fig. 1.
[c]See footnote "c" of Table 3 for α, β, γ, δ.

carboxylate, Fig. 2b [25]. However, ^{119}Sn chemical shift value 132.2 ppm supports the monomeric tetracoordinated geometry around the tin atom in solution, Fig. 2a.

It has been reported that diorganotin dicarboxylates exhibiting ρ value greater than 2.1 possess a trans-octahedral geometry around the tin atom. Thus compound **Ia**, **IIIa**, and **Va** show monomeric trans-R_2SnO_4 hexacoordinated geometry as given in Fig. 2e [23], but δ ^{119}Sn values in noncoordinating solvents advocate a pentacoordinate configuration compatible to the literature [26].

Mass Spectrometry

The mass fragmentation patterns of tri- and diorganotin complexes are shown in Schemes 1 and 2, respectively. A molecular ion peak of reasonable intensity is observed for all the investigated compounds (Tables 8 and 9). In the triorganotin complexes, the primary fragmentation is due to the loss of R group, which appears as a base peak except in compound **VI**. The secondary fragmentation involves loss of CO_2 or $R'CO_2$ group. However, the latter is a more probable pathway.

In diorganotin complexes primary fragmentation involves loss of one $R'CO_2$ group. But if primary fragmentation is due to loss of R group then there is a successive elimination of two CO_2 molecules. The rest of the fragmentation adopts about the same pattern as reported earlier [23]. Dimeric stannoxanes also show similar fragmentation pattern as in the diorganotin complexes except for very weak peaks $[R_2SnO]^+$ and $[RSnO]^+$.

TABLE 7 ^{119m}Sn Mössbauer and ^{119}Sn NMR Data for Organotin(IV) Derivatives of (*E*)-3-(4-Methoxyphenyl)-2-phenyl-2-propenoic Acid[a]

Comp. No.	*IS (mms^{-1})*	*QS (mms^{-1})*	*P = QS/IS*	*^{119}Sn (δ ppm)*
Ia	1.25	3.37	2.696	−136.7
Ib	–	–	–	−174.5, −188.6
II	1.41	3.80	2.695	132.2
IIIa	1.36	3.38	2.485	−165.6
IIIb	–	–	–	−203.7, −226.8
IV	–	–	–	103.9
Va	1.46	3.45	2.363	−170.7
Vb	–	–	–	−210.7, −224.5
VI	–	–	–	−116.8

[a]IS = Isomer shift (mms^{-1}); QS = quadruple splitting (mms^{-1}).

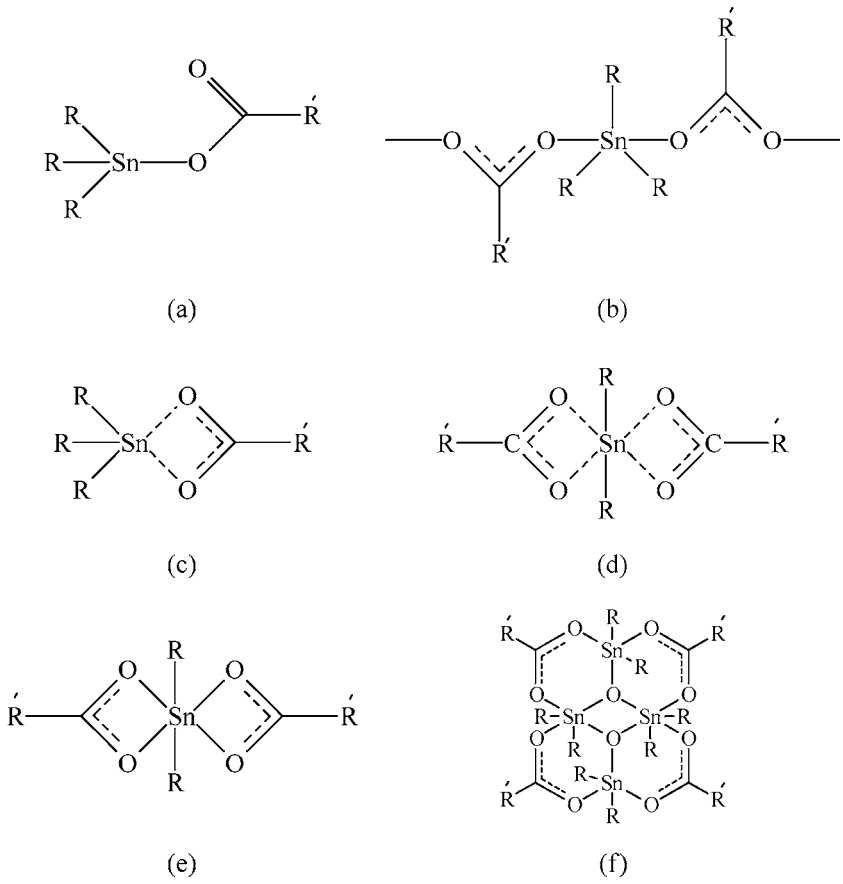
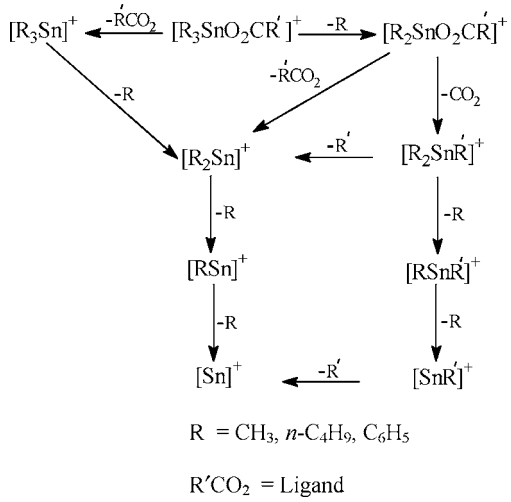


FIGURE 2 Proposed structures (a), (b), (c) for triorganotin(IV) derivatives, (d), (e) for diorganotin(IV) derivatives, and (f) for dimeric tetraorganodicarboxylato stannoxanes.

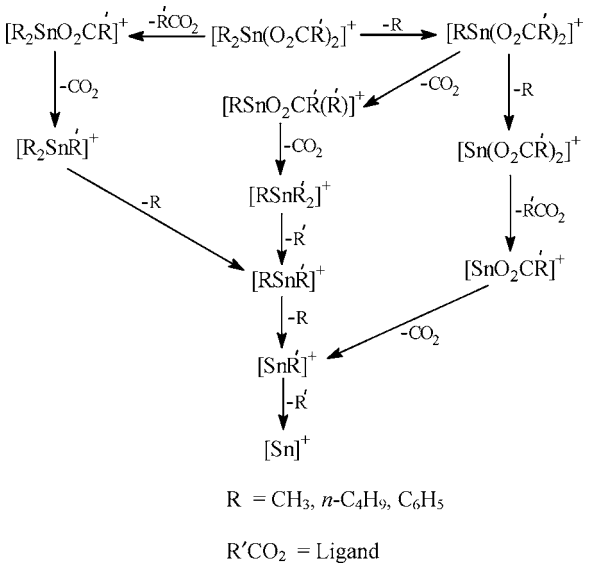
Biological Activity

Biological activity test for some synthesized complexes against various bacteria was carried out by the "agar well diffusion method" and results are summarized in Table 10 [27]. The biological studies of these compounds were also extended to find out their microbial toxicity and cytotoxicity against various fungi and brine-shrimp, respectively as shown in Tables 11 and 12. The screening tests show that the ligand (acid) and compounds **Ia** and **II** are almost inactive. Diorganotin compounds **IIIa** and **Va** have more activity than the respective triorganotin compounds (Table 10). This contradicts the assessment that increase in the number of R groups augments the biological activity of the organotin(IV) carboxylates, as reported earlier [9,28]. These results indicate that anionic ligand may also play an important role in the biocidal activity of organotin compounds.

The antifungal activity of ligand (acid) and its organotin derivatives are given in Table 11, which shows that triorganotin complexes are more active than the diorganotins and ligand itself. Amongst the diorganotin derivatives, diethyltin compound and also ligand acid show high activity only against *Trichophyton longifusus* and *Microsporum canis*. Dimethyltin derivative has no significant



SCHEME 1 Fragmentation pattern of R_3SnL.



SCHEME 2 Fragmentation pattern of R_2SnL_2.

activity while di-*n*-butyltin(IV) complex is completely inactive.

The cytotoxicity data have been collected by the brine-shrimp lethality bioassay method [29] and results are given in Table 12, which indicate that compounds **Ia** and **IIIa** are nontoxic. However, all other complexes showed cytotoxicity with LD_{50} values in the range 0.2584–71.6493 μg/mL. Thus, compound **Va** has been found to be least toxic and **VI** was the most toxic of all the synthesized organotin(IV) derivatives of (*E*)-3-(4-methoxyphenyl)-2-phenyl-2-propenoic acid.

EXPERIMENTAL

Chemicals and Instrumentation

All chemicals were of analytical grade and used without further purification. The ligand (*E*)-3-(4-methoxyphenyl)-2-phenyl-2-propenoic acid was prepared in laboratory [30]. Solvents were dried in situ using standard methods [31].

Melting points were determined on a MPD Mitamura Riken Kogyo (Japan) electrothermal melting point apparatus and are uncorrected. Infrared spectra were recorded as KBr disks on a Bio-Rad *Excaliber* FT-IR, model FTS 300 MX spectrometer (USA). ^{1}H and proton decoupled ^{13}C and ^{119}Sn NMR spectra were taken (in $CDCl_3$ (40%) at 298 K) on a Bruker ARX 250 FT-NMR instrument, using $CDCl_3$ as internal reference [δ ^{1}H($CDCl_3$ = 7.25: δ ^{13}C = 77.0)]. ^{119}Sn NMR spectra were obtained with Me_4Sn as an external reference [Ξ (Sn) = 37.290665] [32]. The mass spectral data were measured on a MAT-8500 Finnigan (Germany) mass spectrometer. ^{119m}Sn Mössbauer spectra were obtained with a constant acceleration on a ranger (model MS-900 Mössbauer, USA) spectrometer. A barium stannate source was used at room temperature; samples were mounted in Teflon holders and cooled to 80 K using a liquid nitrogen cryostat. The resulted spectra were analyzed by a least-square fit to Lorenzian shaped lines.

General Procedure for Synthesis

The sodium salt of (*E*)-3-(4-methoxyphenyl)-2-phenyl-2-propenoic acid was prepared by dissolving 4.5 g, 16.3 mmol of ligand in ethanol (150 mL), followed by addition of 1.4 g, 16.3 mmol of sodium bicarbonate as an aqueous solution. The mixture was allowed to stir for 2 h at room temperature and then evaporated under vacuum. The resultant solid so obtained was dried (mp = 275–277°C).

Stoichiometric amount of sodium salt of (*E*)-3-(4-methoxyphenyl)-2-phenyl-2-propenoic acid (2.0 g, 7.25 mmol) with diorganotin dichloride (3.63 mmol) and triorganotin chloride (7.25 mmol) was refluxed in dry toluene (80 mL) for 8–10 h in

TABLE 8 Fragmentation Pattern and Relative Abundance of Triorganotin(IV) Derivatives Observed at 70 eV

Fragment Ions	(**II**) *[m/z (%)] R = CH_3*	(**IV**) *[m/z (%)] R = n-C_4H_9*	(**VI**) *[m/z (%)] R = C_6H_5*
$[R_3SnO_2CR']^+$	418 (14)	544 (1)	604 (24)
$[R_2SnO_2CR']^+$	403 (100)	487 (100)	527 (39)
$[R_2SnR']^+$	359 (95)	443 (3)	483 (21)
$[RSnR']^+$	344 (0.6)	386 (5)	–
$[R_3Sn]^+$	165 (30)	291 (1)	351 (100)
$[R_2Sn]^+$	150 (5)	234 (2)	274 (16)
$[RSn]^+$	135 (5)	177 (7)	197 (35)
$[SnR']^+$	329 (78)	329 (16)	–
$[Sn]^+$	120 (2)	120 (4)	120 (16)
$[R'COOH]^+$	254 (2)	254 (14)	254 (7)
R′	209 (77)	209 (21)	209 (20)

R′ = C_6H_5CCH ($C_6H_4OCH_3$).

TABLE 9 Fragmentation Pattern and Relative Abundance of Diorganotin(IV) Derivatives Observed at 70 eV

Fragmentation Ions	(**I**) *[m/z (%)] R = CH_3*	(**IIIa**) *[m/z (%)] R = n-C_4H_9*	(**V**) *[m/z (%)] R = C_2H_5*
$[R_2Sn(O_2CR')_2]^+$	656 (1)	740 (2)	684 (0.6)
$[R_2SnO_2CR']^+$	403 (86)	487 (100)	431 (62)
$[RSn(O_2CR')_2]^+$	641 (2)	683 (92)	655 (100)
$[RSnO_2CR'(R')]^+$	597 (1)	639 (8)	611 (8)
$[RSnR'_2]^+$	553 (0.2)	595 (1)	567 (2)
$[R_2SnR']^+$	359 (100)	443 (2)	387 (9)
$[RSnR']^+$	344 (0.5)	386 (5)	358 (4)
$[Sn(O_2CR')_2]^+$	–	626 (2)	–
$[SnO_2CR']^+$	–	373 (9)	–
$[SnR']^+$	329 (6)	329 (45)	329 (55)
$[Sn]^+$	120 (3)	120 (2)	120 (2)
$[R'CO_2H]^+$	254 (19)	254 (6)	254 (7)
R′	209 (79)	209 (92)	209 (91)

R′ = $C_6H_5CCH(C_6H_4OCH_3)$.

TABLE 10 Bactericidal[a,b] Data of Organotin(IV) Derivatives of (*E*)-3-(4-Methoxyphenyl)-2-phenyl-2-propenoic Acid

		Zone of Inhibition (mm)							
Name of Bacterium	*Clinical Implication*	(**Ia**)	(**II**)	(**IIIa**)	(**IV**)	(**Va**)	(**VI**)	*Acid*	*Ref. Drug*
Escherichia coli	Infection of wounds, urinary tract and dysentery	–	–	14	–	13	–	–	30
Bacillus subtilis	Food poisoning	–	–	17	24	15	–	–	31
Shigella flexenari	Blood diarrhea with fever and severe prostration	–	–	14	14	12	13	–	32
Staphlococcus aureus	Food poisoning, scaled skin syndrome, endocarditis	–	13	14	–	13	12	–	43
Pseudomonas aeruginosa	Infection of wounds, eyes, septicemia	–	–	15	14	12	11	–	25
Salmonella typhi	Typhoid fever, localized infection	–	–	16	15	13	20	–	41

[a]In vitro, agar well diffusion method, conc. 1 mg/mL of DMSO.
[b]Reference drug, Imipenum.

TABLE 11 Antifungal[a] Activity of Organotin(IV) Derivatives of (*E*)-3-(4-Methoxyphenyl)-2-phenyl-2-propenoic Acid

	Percent Inhibition									
Name of Fungus	(**Ia**)	(**II**)	(**IIIa**)	(**IV**)	(**Va**)	(**VI**)	**Acid**	*Standard Drug*	*Percent Inhibition*	*MIC[b] (μg/mL)*
Trichophyton longifusus	25	95	–	90	80	88	70	Miconazole	100	70
Candida albicans	–	90	–	100	–	85	–	Miconazole	100	110.8
Aspergillus flavus	20	95	–	88	–	90	–	Amphotericin B	100	20
Microsporum canis	20	90	–	90	83	88	60	Miconazole	100	98.4
Fusarium solani	–	90	–	90	–	80	–	Miconazole	100	73.25
Candida glaberata	–	–	–	80	–	75	–	Miconazole	100	110.8

[a]Concentration: 200 μg/mL of media.
[b]MIC = Minimum inhibitory concentration.

TABLE 12 Cytotoxicity[a,b] Data of Organotin(IV) Derivatives of (*E*)-3-(4-Methoxyphenyl)-2-phenyl-2-propenoic Acid

Comp.	(**Ia**)	(**II**)	(**IIIa**)	(**IV**)	(**Va**)	(**VI**)	Acid
LD_{50}	–	1.2660	–	0.4254	71.6493	0.2584	–

[a]Against Brine-Shrimps (in vitro).
[b]No cytotoxicity for compounds (**Ia**), (**IIIa**), and **acid**.

a two-necked round bottom flask equipped with all accessories. It was placed overnight at room temperature and then filtered. Solvent was rotary evaporated, and solid obtained was recrystallized from a mixture of chloroform: *n*-hexane (2:1).

The dimeric distannoxanes (**Ib**, **IIIb**, and **Vb**) were synthesized by refluxing mixture of the (*E*)-3-(4-methoxyphenyl)-2-phenyl-2-propenoic acid and corresponding diorganotin oxide (1:1) in dry toluene (90 mL) for 8–10 h, using Dean and Stark apparatus for continuous removal of water formed during condensation reaction. The rest of procedure adopted was same as given above.

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